

Welcome to the College of Microscopy!

Food and beverages are not permitted in the auditorium, please enjoy them in the Atrium or Café.

Power receptacles are located between every other seat for your convenience.

Microanalysis of Particles

a

Microbeam Analysis Society

Topical Conference

Hosted by the

College of Microscopy

Westmont, IL

April 20-23, 2009



Welcome!



Introduction

In case of emergency...

Overview of Conference Logistics

Thank you to Sponsors for their support and participation

The Program

Microanalysis of Particles: Summary

Conference Format

Mornings and immediately after lunch will be devoted to lectures here in the auditorium.

During the morning coffee break please be sure to sign up for two of the workshops. They're repeated so don't worry...

For the afternoon workshops, please seek out an escort holding a sign with the session name you selected and follow them to the session.

Lunch will be brought in so that our schedule can remain on track.

Posters are up for the duration, please examine them at your leisure, or during the afternoon coffee break/poster sessions.

The Banquet tonight: buses will take us to ANL and return us here. Be prepared for the bus to leave at 4:30.

The buses will bring us back after our tour of the APS.

Tomorrow evening stick around or come back for an evening with EDAX.



IXRF



M³S

Thank you to the Generosity of our Sponsors

Katherine L. Crispin: Case Western Reserve University

Phillip Gopon: University of Wisconsin – Madison

Mark A. Sutcliffe: University of Missouri - Columbia

Nicholas A. Wright: Milliken University

Microanalysis of Particles: An Overview

From the “basics” to the latest and greatest...

Sample isolation and preparation

Best practices for various methods

Theoretical concepts to keep in mind

Use of automation and simulation for improved sample characterization

Utilizing new instrumentation to chart new territory

What is a particle?

The definition depends on one's scientific specialization. The size ranges used by atmospheric scientists covers the primary size ranges.

In ecology it might be defined as a small object.

In nanotechnology it is defined as a small object that behaves as a whole unit in terms of its transport and its properties.

Fraction	Size range
PM ₁₀ (thoracic fraction)	≤10 μm
PM _{2.5} (respirable fraction)	≤2.5 μm
PM ₁	≤1 μm
Ultrafine (UFP or UP)	≤0.1 μm
PM ₁₀ -PM _{2.5} (coarse fraction)	2.5 μm - 10 μm

Particles are contaminants, flaws in emulsions or coatings, thin film inconsistencies, fabrication failures, mysterious powders or something else, particles are the root cause of many industrial, environmental, and national security concerns.

What is that particle made of?

Enter the realm of microanalysis!

Enter the realm of nanoanalysis!

Complementary Analytical Approach

Electron beam techniques

Mass spectrometry techniques

X-ray diffraction

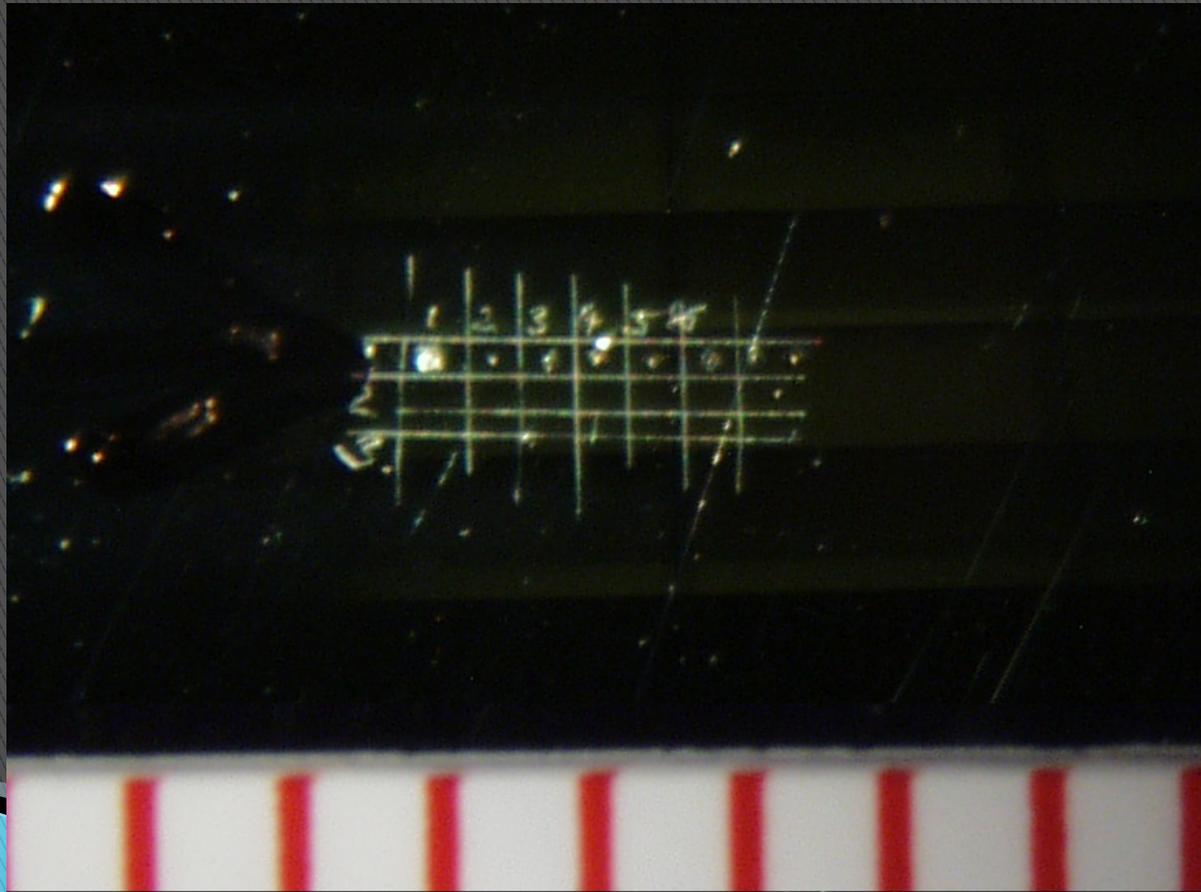
Synchrotron techniques

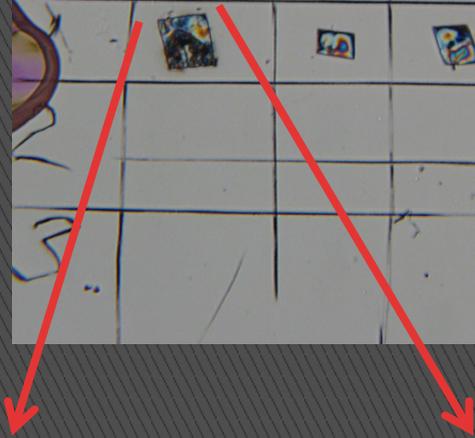
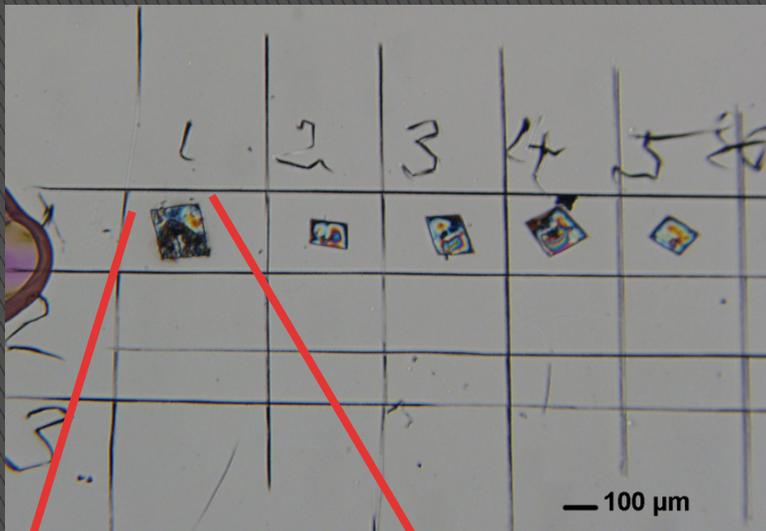
Micro X-ray techniques

Simulation tools

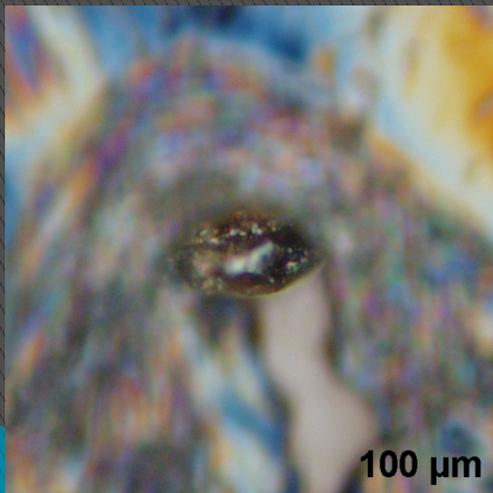
Sample isolation and preparation the key to successful analysis

Optical microscopy

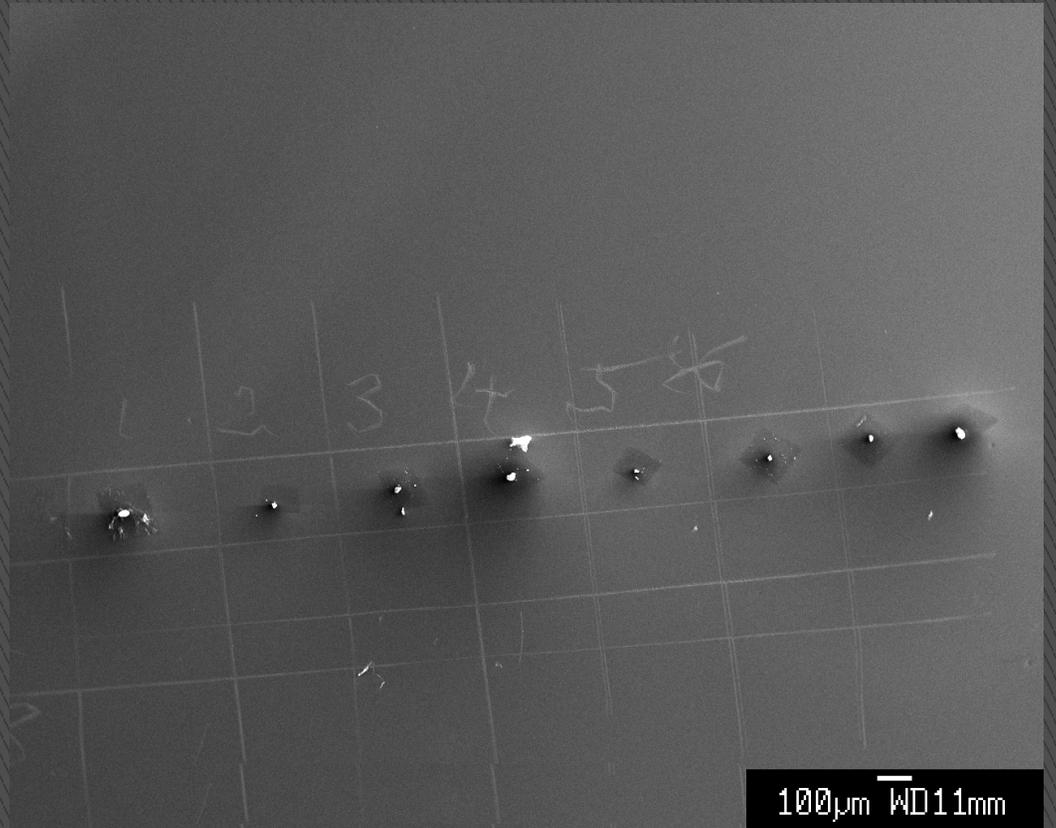




5X obj



50X obj



SEI

Some techniques require additional, highly specialized sample preparation methods.

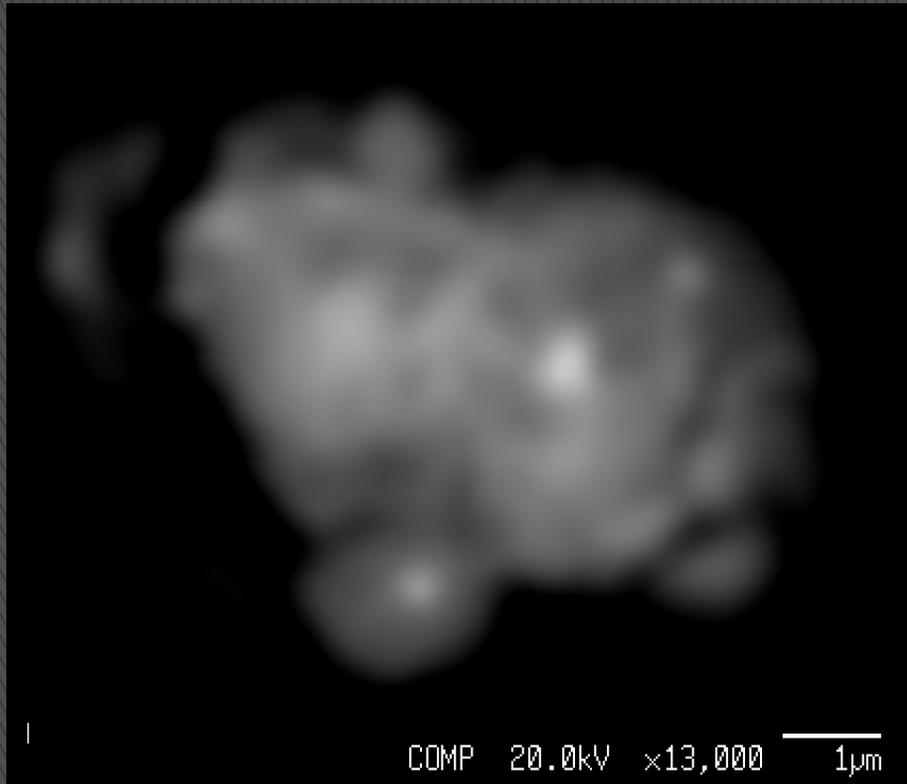
For example TEM/AEM require samples, even particles to be sub-sampled in order to provide successful results.

FIB-SEM instruments provide very exciting opportunities for sample preparation as well as even potentially more incredible opportunities for in place milling and sample characterization.

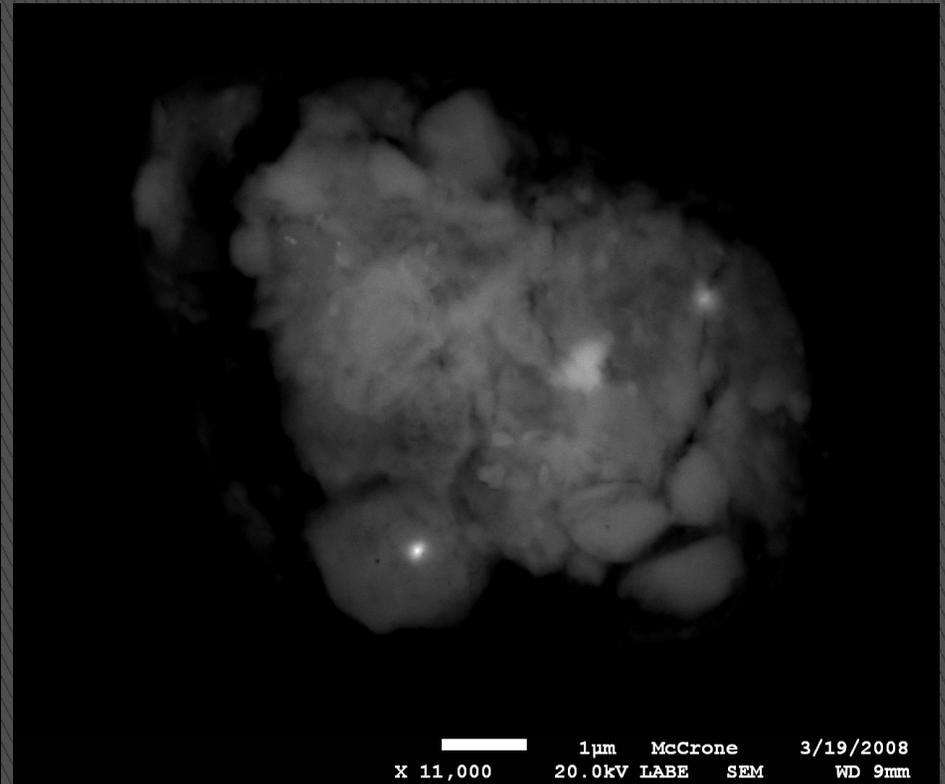
New bench top equipment for sample preparation that is much more economically achievable.

We'll hear about the latest efforts to apply new "extreme" geometry corrections to the cadre of quantitative reduction schemes. Plus we'll learn how to utilize cutting edge simulation tools to test whether the information we measure matches what we think we know about it, or is that the other way around...

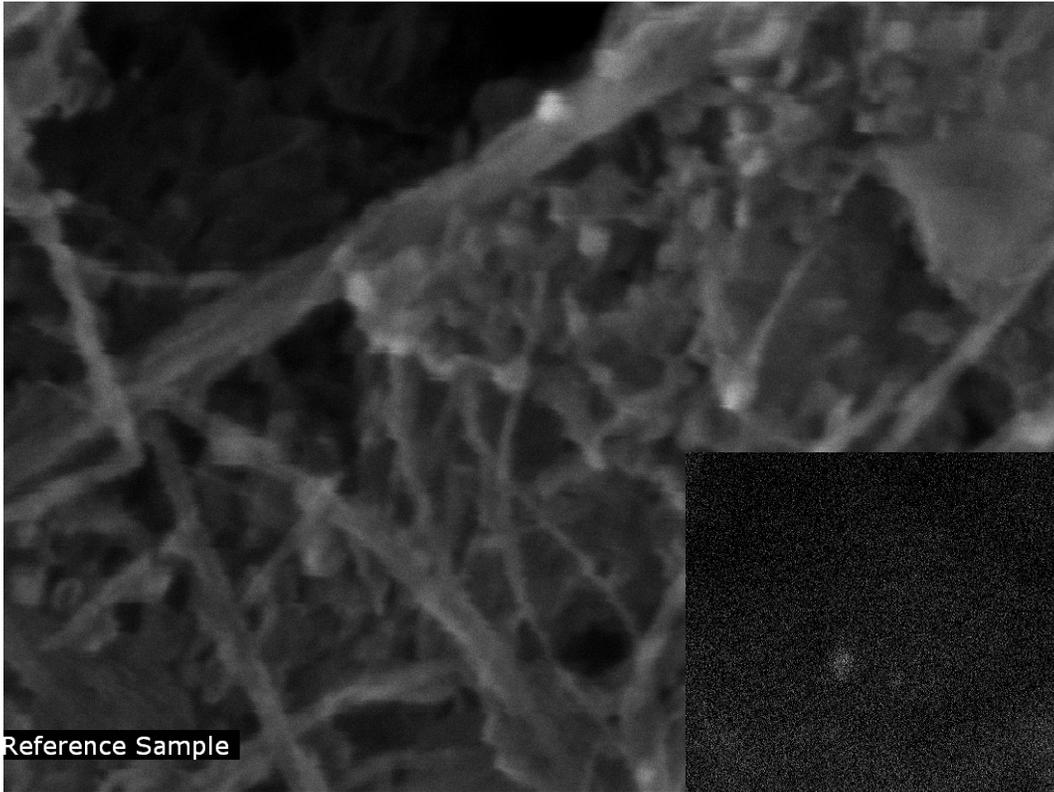
Generational comparison



JXA-8200



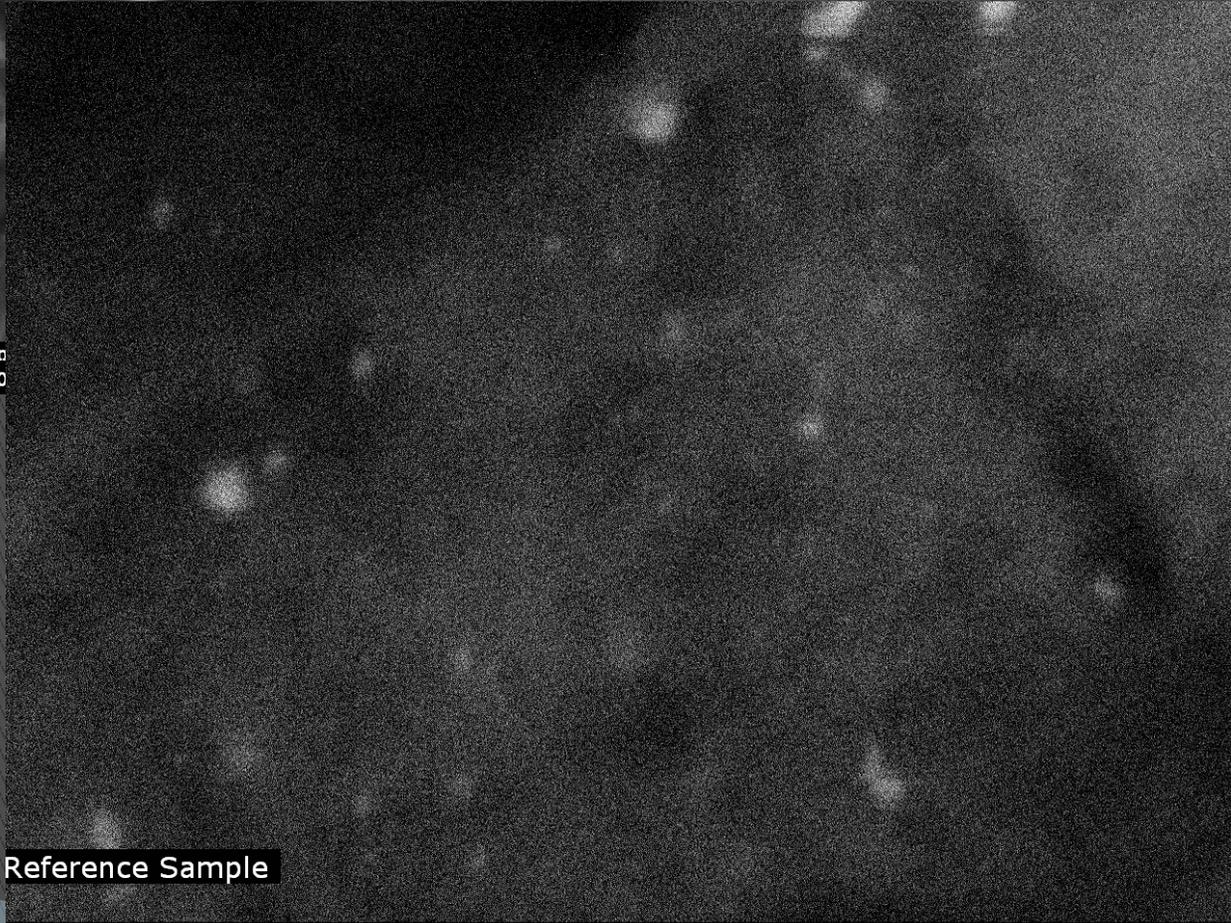
JSM-7500F



Reference Sample

10nm
X 300,000

5 kV
Composition of the catalyst particles identified by EDS.



Reference Sample

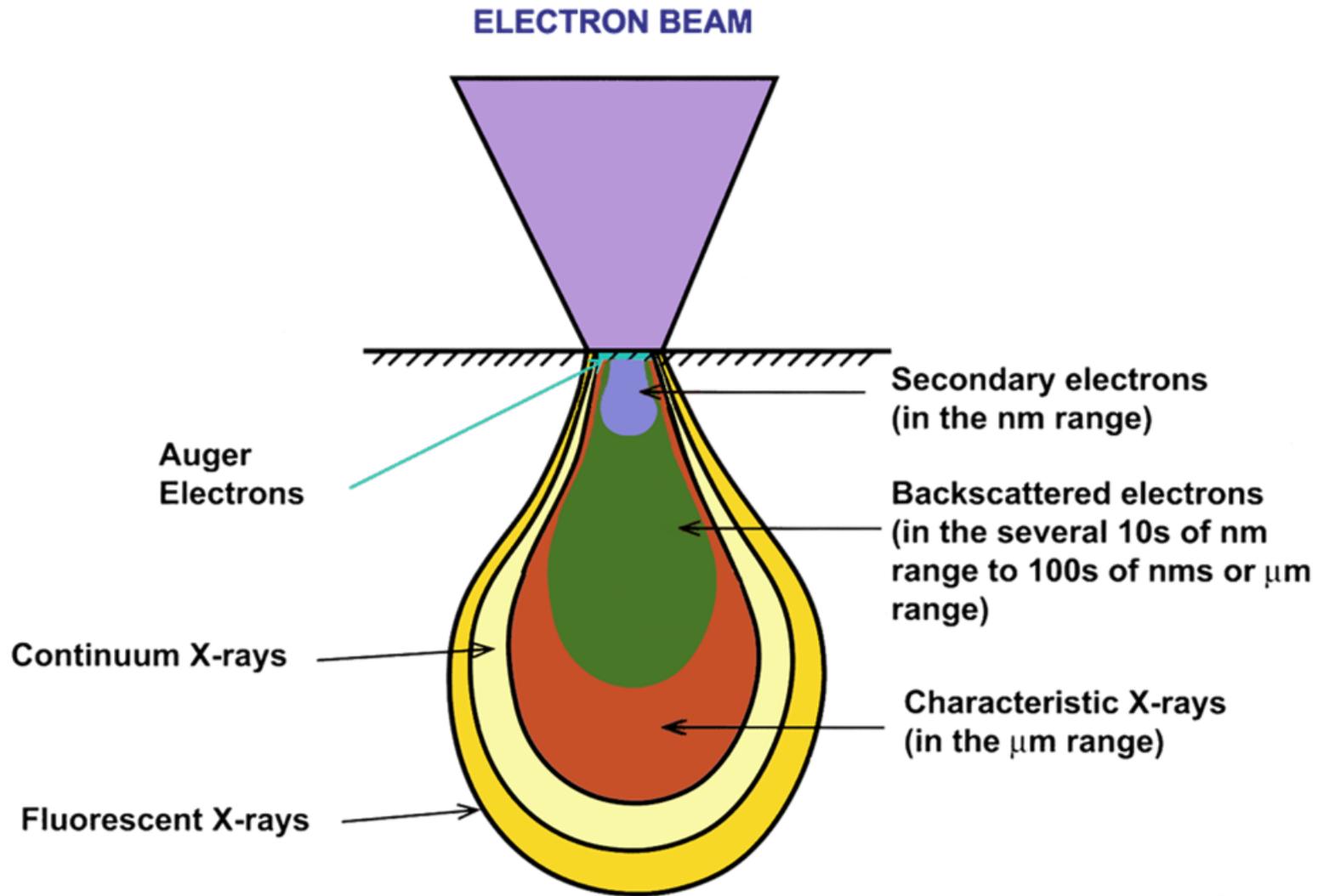
10nm
X 300,000 5.0kV LAGE WD 4mm 9/5/2008

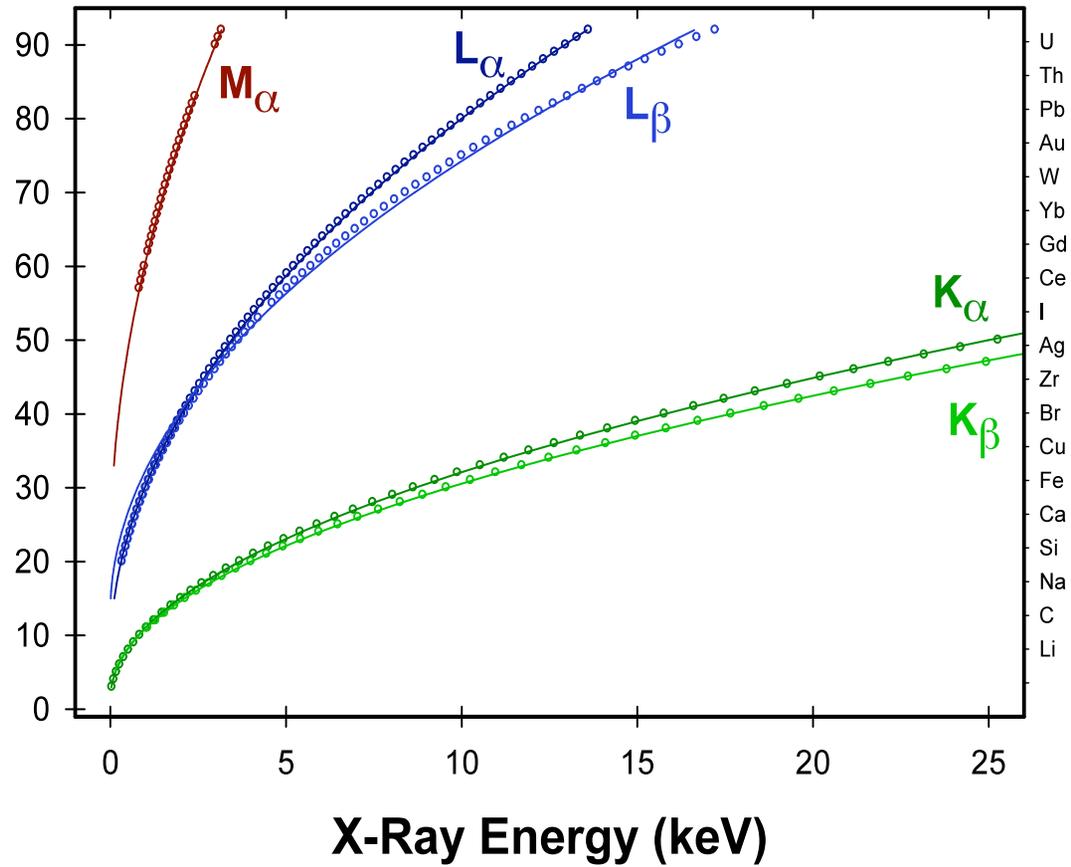
We'll hear about efforts to compensate for the geometric effects, we'll see the latest efforts obtained additional latest generation electron microscopy equipment, learn about obtaining crystal structure information from particles around 10 micrometers in diameter, and learn about cool new bright source techniques at the APS.

Lou Ross extends his greetings and his apologies.

Qualitative EDS: Understanding the basics

Concepts to keep in mind





$$\sqrt{E} = C_1 \times (Z - C_2)$$

C_1, C_2 are constants

Two more major factors which influence X-rays emitted from sample:

X-ray Absorption (A): *photoelectric effect*: ionization of inner shell electrons.

Described by **Beer's Law**:

μ/ρ = mass absorption coefficient

$$I = I_0 \times e^{-(\mu/\rho) \cdot \rho t}$$

= density of material

= thickness (expon. dependence)

jumps in μ/ρ at critical ionization energies in sample: **X-ray absorption edges**

Secondary X-ray Fluorescence (F): production of secondary X-rays from ionization from X-ray absorption:

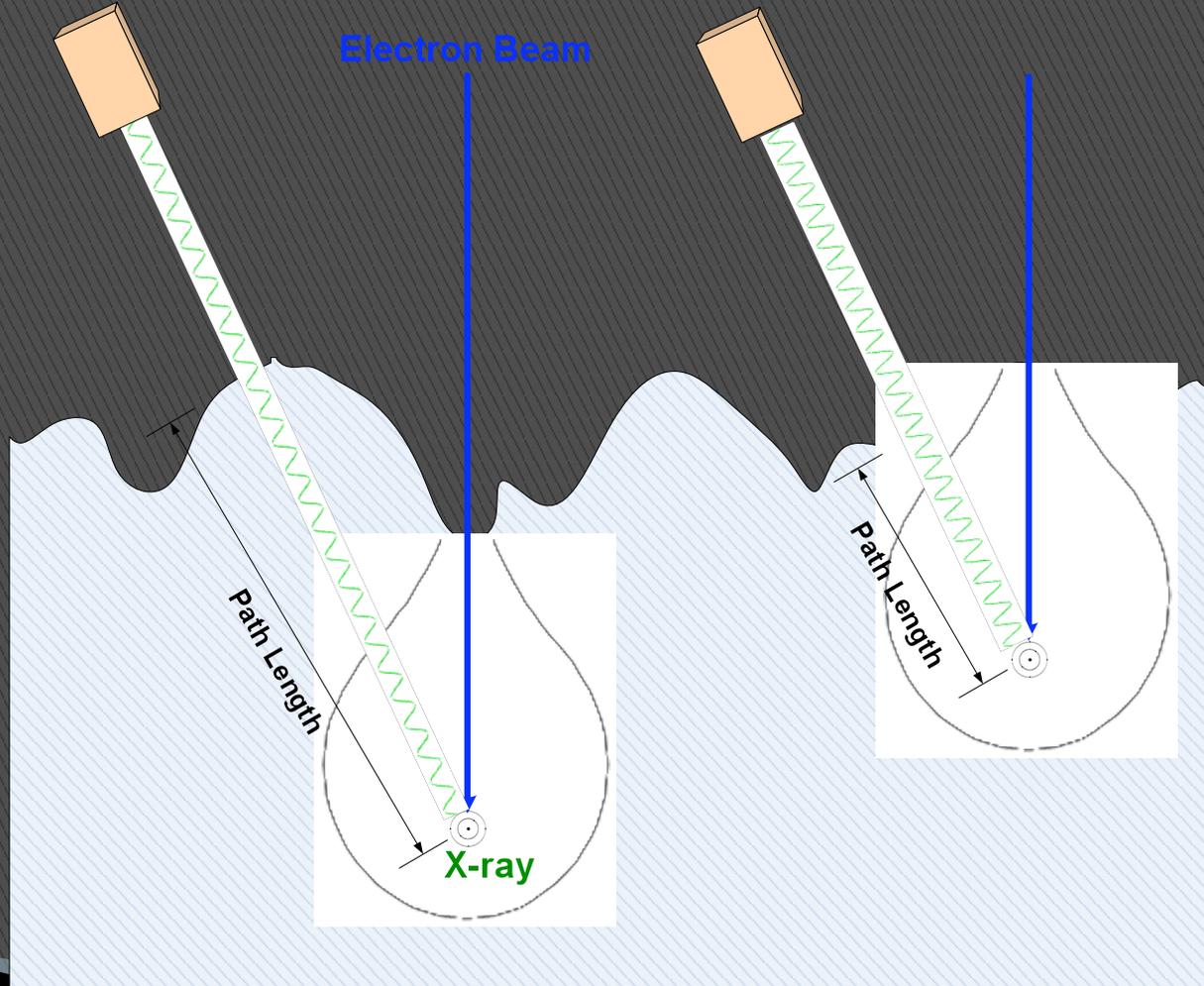
Area of secondary fluorescence can be larger than electron interaction volume, since X-rays are more penetrating than electrons.

Can be produced by characteristic and continuum X-rays

Absorption is not constant for samples with geometry.

X-ray Detector

Electron Beam



Path Length

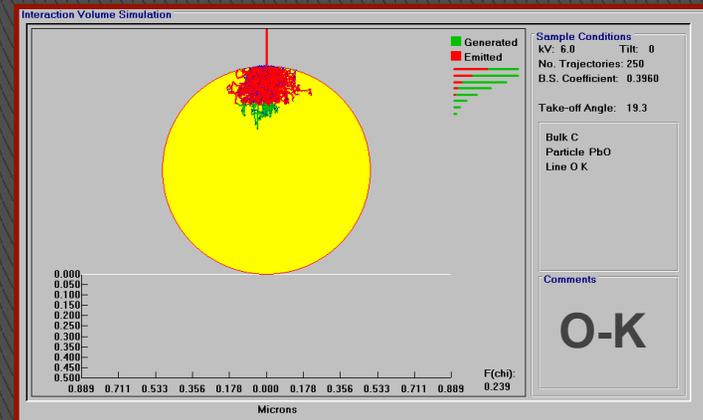
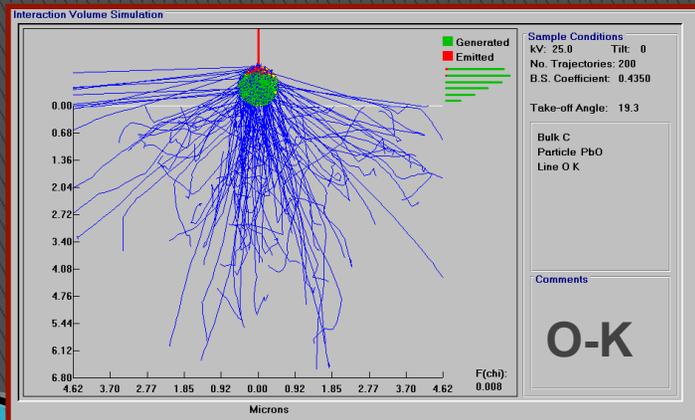
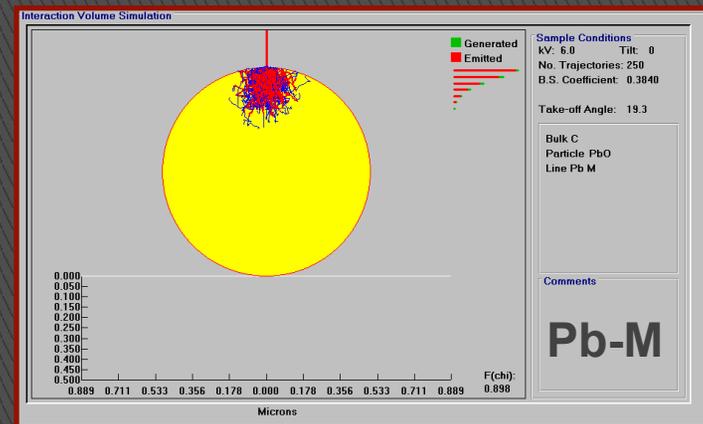
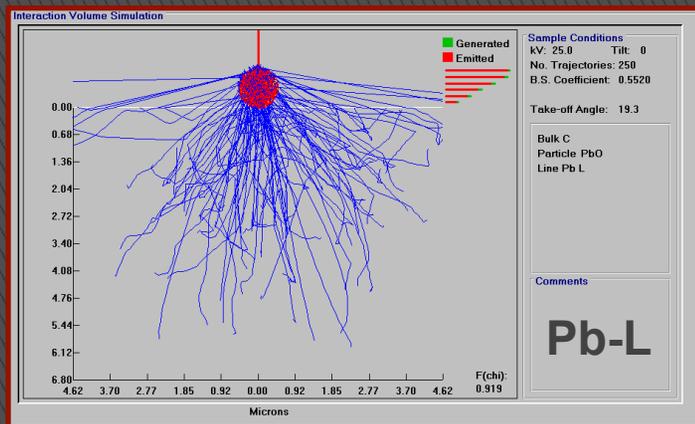
Path Length

X-ray

Analysis of a PbO particle 1 μm in diameter.

25kV

6 kV

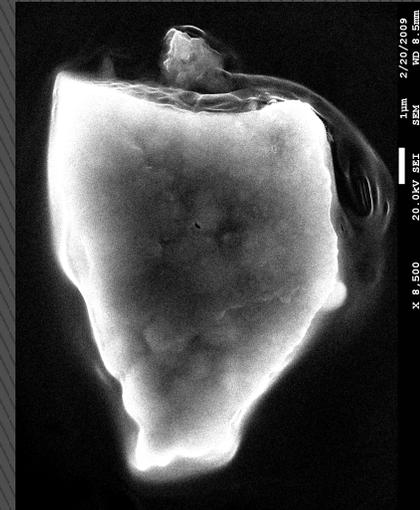
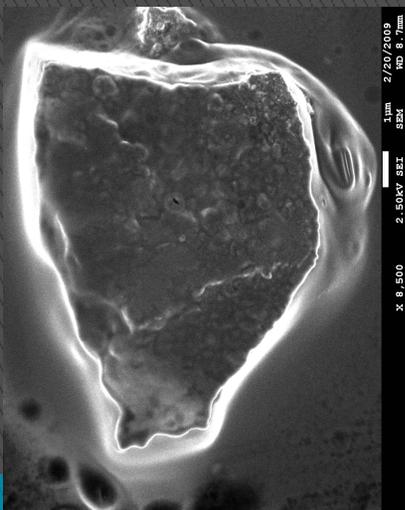
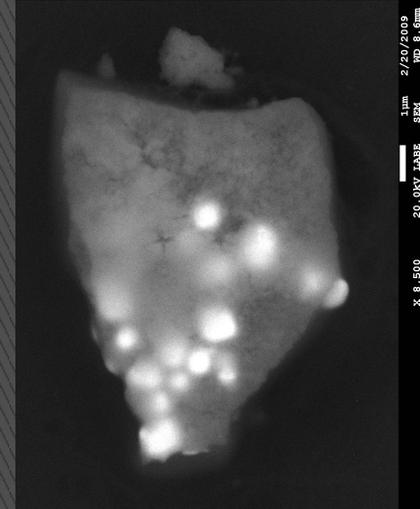
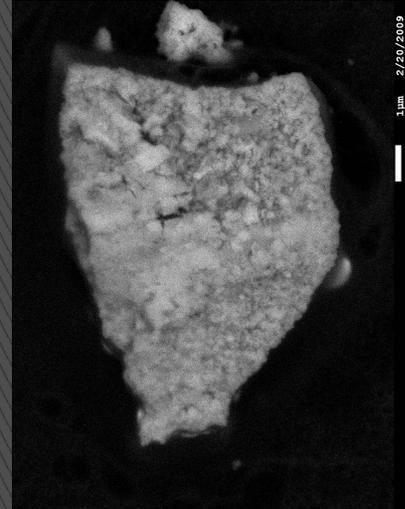
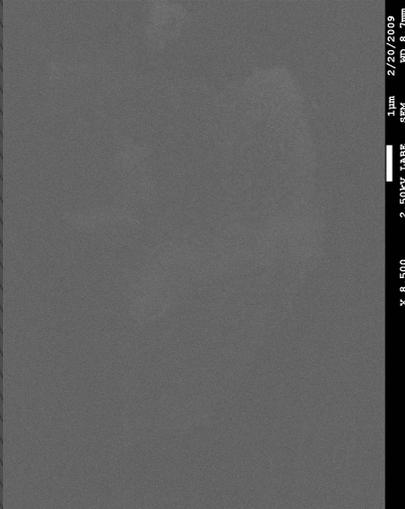


The interaction volumes producing the various electrons and X radiation are different for each atom in the sample.

- Electron beam energy determines the interaction volume of sample
- Characteristic X-rays are produced by ionization of atoms in sample
- Energy of characteristic X-rays used for qualitative analysis
- Intensity of characteristic X-rays affected by operating conditions and sample characteristics (**matrix effects**)
- Intensity of characteristic x-rays affected by atomic number (**Z**), absorption (**A**), secondary fluorescence (**F**)
- Intensities of X-ray lines can be used for quantitative analysis **provided one has similar standard reference materials**
- For particles, geometry and size have huge influence

An electron imaging example of the Basics

BEI



SEI 2.5 kV

5 kV

10 kV

20 kV

**Thank you for your participation,
thank you to the speakers, and
thank you to our sponsors and host!**